THIN FILM THERMOPHYSICAL PROPERTY CHARACTERIZATION BY SCANNING LASER THERMOELECTRIC MICROSCOPE¹

G. Chen^{2, 3} and T. Borca-Tasciuc²

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² Department of Mechanical Engineering and Materials Science, Duke University, Durham, NC 27708.

³ To whom all correspondence should be addressed.

ABSTRACT

This work presents a scanning laser based thermal diffusivity measurement technique

for thin films as well as bulk materials. In this technique, a modulated laser beam is focused

through a transparent substrate onto the film-substrate interface. The generated thermal

wave is detected using a fast responding thermocouple formed between the sample surface

and the tip of a sharp probe. By scanning the laser beam around the thermocouple, the

amplitude and phase distributions of the thermal wave are obtained with micrometer

resolution. Thermal diffusivity of the film is determined by fitting the obtained phase signal

with a three dimensional heat conduction model. Experimental results are presented for a

150nm gold film evaporated on a glass substrate.

KEY WORDS: heat conduction; photothermal method; scanning laser; thermal conductivity;

thermal diffusivity; thermoelectric effect; thin film.

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1. INTRODUCTION

Measurement of the thermophysical properties of thin films is a challenging task and many techniques have been developed. Available techniques and their range of applications have been summarized in several publications [1-4]. To determine the in-plane thermal conductivity or thermal diffusivity of thin films, substrate removal is often necessary. Alternative thin film thermophysical property measurement techniques, particularly methods that are capable of determining anisotropic thermophysical properties of thin films without removing the substrate, will provide valuable tools for understanding heat transfer in thin films and for exploring thin film based devices.

The difficulty in determining the in-plane thermophysical properties of thin films lies in the fact that most heat is conducted through the substrate rather then through the film. One possible way to overcome this difficulty is to obtain two dimensional temperature distribution around a microscale heat source. From the spread of the heat source, it may be possible to obtain anisotropic thermophysical properties of the thin film and/or the bulk material. One technique that gives the two-dimensional thermal field of a thin film on substrate structure is an optical pump-and-probe method by scanning either the pump laser or the probe laser [5-7]. The disadvantage of this method is in the difficulty of aligning two laser beams through the focusing optics. Kemp et al. [8] developed a method that maps the temperature distribution of a free-standing film generated by a scanning laser and detected by a thermocouple formed by welding a fine bismuth-antimony-telluride probe to the sample.

This paper extends the idea of Kemp et al. [8] for free-standing thin films and develops a method suitable for determining anisotropic thermophysical properties of thin films for film-on-substrate systems without the removal of the substrate. The method employs modulated laser radiation to generate a thermal wave in the sample, and scans the focused laser beam over the area of interest. The laser wavelength is tuned to a range where the

substrate is transparent such that the laser beam penetrates the substrate and acts as a micrometer scale heat source in the film. The detection is done using a fast thermoelectric effect created by direct contact between the metallically covered surface of the sample and the sharp tip of an electrochemically etched wire. Thermophysical properties of the film or the substrate material can be determined from the amplitude and/or the phase of the thermoelectric voltage signal.

2. EXPERIMENT

The method is designed to measure thermophysical properties of films on a substrate or bulk materials. A focused laser beam is scanned through the transparent substrate over the film-substrate interface in order to produce thermal waves that are detected using a fine thermocouple formed between the sample and the probe. Thermophysical properties can be determined from the amplitude and phase profiles of the data. Our current experimental setup is shown in Fig.1.4 An argon laser is used to pump a tunable Ti-Sapphire laser that may output laser radiation with a wavelength between 700-900 nm. The modulated radiation is coupled into a single mode optical fiber. The optical system used to focused the laser radiation is assembled onto a base plate, which is placed onto a x-y scanner (in our case, it is fitted into the scanner of an atomic force microscope). A specially designed holder is used to maintain the sample at an adjustable distance above the scanning laser such that radiation penetrates the transparent sample and is focused on the top surface. The top surface is a conducting element that absorbs the laser radiation and also forms one leg of the thermocouple. The other leg of the thermocouple is formed by a fine wire contacting the

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⁴ A diode laser is preferable to the Ti-Sapphire laser for a compact system.

surface. The thermocouple junction formed between the film and the wire has virtually no mass and is thus expected to be fast responding.

An experiment is conducted with a 150nm gold film evaporated on the top surface of a glass slide. The probe used to detect the thermal wave is a sharp K-type thermocouple wire obtained through electrochemical etching in a solution of sulfuric acid. The tip is brought into contact with the gold film. The ac components of the amplitude and the phase of the thermoelectric voltage developed at the tip-surface junction are picked-up by a lock-in amplifier.

The laser was scanned over a $128\mu\text{m}\times128\mu\text{m}$ area around the probe. The minimum laser beam diameter as estimated using the ABCD law is about $1~\mu\text{m}$ [9]. At a scan size of $128\mu\text{m}$ and a round-trip frequency of 0.1 Hz (the minimum frequency rate in the current setup), the scanning speed of the laser is $u=25.6\times10^{-6}\text{m/s}$. The maximum modulation frequency of our current set-up is 3200~Hz. Under these conditions, the ac temperature detected by the stationary probe as a function of the laser beam location is equivalent to the ac temperature profile around a fixed heat source [10]. For a film-on-substrate structure being heated at the film-substrate interface, assuming that the film is thermally thin and optically thick, the ac temperature rise on the surface is given by the following expression that contains both the amplitude and the phase information of the temperature profile [10]:

$$\theta_{fAC}(r) = \frac{I_0 R^2 \int_0^\infty \beta J_0(\beta r) e^{-R^2 \beta^2 / 4} d\beta}{2 \int_0^\infty Z_f \sinh(m_f d) + Z_s \cosh(m_f d)}$$
(1)

where θ_{fAC} is the ac temperature rise on the film surface, r the radial coordinate, I_0 the absorbed laser beam intensity, R the radius of the laser beam, β the integration variable, J_0 the Bessel function of order zero, d is the film thickness. Other quantities in Eq.(1) are defined as:

$$Z_{s} = k_{s} m_{s}, Z_{f} = k_{fz} m_{f}$$
 (2)

$$m_s = \sqrt{\beta^2 - i\omega/\alpha_s} \tag{3}$$

$$m_f = \sqrt{\gamma \beta^2 - i\omega / \alpha_{fz}} \tag{4}$$

where α_s is the thermal diffusivity of the substrate and α_{fz} the cross-plane thermal diffusivity of the film, k_s and k_{fz} are the corresponding thermal conductivity, ω the angular modulation frequency, and γ is the ratio of in-plane to cross-plane thermal conductivity of the film.

Based on Eq.(1) it is possible to determine both the in-plane and the cross-plane thermal diffusivities, depending on which is the dominant heat conduction direction. The latter is influenced by several factors, including film and substrate thermal conductivities, modulation frequency, laser beam radius and location of detection. In the present experimental technique several of these factors can be controlled and thus used to facilitate the determination of the thermophysical properties. For a highly thermal conductive film at low modulation frequencies heat conduction inside the film is mainly in the in-plane direction and the anisotropy effect is negligible. The in-plane thermal conductivity can be determined from the radial distribution of the phase profile [7]. At high modulation frequencies the thermal wave is confined in a region close to the heat source. If the laser beam diameter is large, the heat flowing in the parallel direction is negligible comparing to the heat flowing perpendicular to the film. Determination of cross-plane thermal conductivity is then possible from the frequency dependence of the thermal signal collected at the beam center.

One other parameter that can affect the determination of the thermophysical properties for a film-on-substrate system is the interface thermal resistance. The effect of the interface thermal resistance on the determination of film thermal diffusivity, was ruled out for this experiment based on a simplified model [7].

3. RESULTS AND DISCUSSION

Figure 2(a) shows the distribution of phase and Fig. 2(b) the distribution of amplitude of the experimental thermal signal collected at the surface of a 150nm gold film evaporated on a glass substrate. The phase signal has a region of linear dependence on the laser beam displacement. This is in agreement with recently reported photothermal reflectance detection method [7].

To obtain the thermal diffusivity of the film, the following fitting scheme is employed. First the slope of the experimental phase signal in the linear region is determined. This slope is then compared with theoretically calculated slope of the phase signal using Eq.(1) for a guessed value of the thermal diffusivity of the film. In these calculations, the thermophysical properties of the window glass are assumed for the substrate [11]. For given film and substrate thermophysical properties, numerical calculations show that the slope of the phase is independent of the laser beam size, as can be seen from Fig. 3.. This behavior makes it possible to find the thermophysical properties of the film even if the laser beam diameter is not exactly known. Assuming a film diffusivity of 0.96 cm²s⁻¹, the theoretical slope of the phase match the experimentally determined slope. Furthermore the laser beam radius may be determined by fitting the experimental phase and normalized amplitude profiles with the theoretical calculations. For the current experiment, heat conduction inside the film is mainly in the in-plane direction and the determined thermal diffusivity is thus in this direction.

Figure 4(a) shows the phase fit and figure 4(b) the amplitude fit of the experimental signal for a thermal diffusivity of the film of 0.96 cm² s⁻¹. The determined laser beam radius for this fit is 2μm, which is close to the calculated 1μm radius based on ABCD law [9]. Hartmann et al. [7], reported a thermal diffusivity of 0.6 cm² s⁻¹ for a 160nm sputtered gold film on quartz.

Our first experiment was performed using a 3 ms time constant for the lock-in amplifier. In order to collect a less noisier signal another run has been done using a 30ms time constant. However the thermal diffusivity value for the film derived from the data collected

under this condition is 0.76 cm² s⁻¹. The discrepancy is thought due to the insufficient stay time at each data point (about 39ms) with the 30 ms time constant.

4. CONCLUSION

This work has demonstrated the potential of the scanning laser thermoelectric microscope for characterizing the thermophysical properties of thin films at micrometer scale without removing the substrate. The microscope employs a modulated, focused laser as the heating source and a fast thermoelectric effect between the sample and a fine probe to detect the temperature response. Phase and amplitude images are presented for laser heating of a 150nm gold film evaporated on a glass substrate. Thermal diffusivity of the film is obtained by fitting these signals with a three-dimensional heat conduction model.

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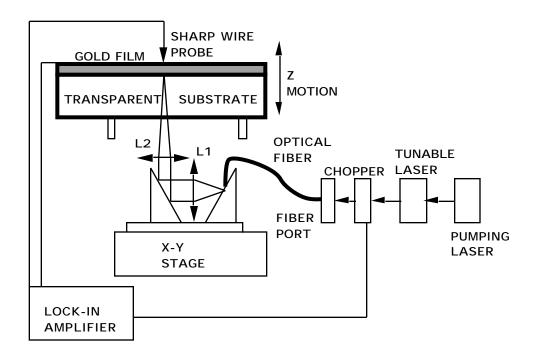
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FIGURE CAPTIONS

- Fig. 1. Schematic drawing of the experimental set-up.
- Fig. 2. Distribution of (a) phase and (b) amplitude of the thermal signal.
- Fig. 3. Theoretically calculated phase signals showing that slope in the linear region is independent of the laser beam radius.
- Fig. 4. Fitting of (a) phase and (b) amplitude of the experimental signal of a gold-on-glass sample with a film thermal diffusivity of 0.96 cm²s⁻¹.



G. Chen and T. Borca-Tasciuc, Fig. 1

